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INTERDISCIPLINARY RESEARCH ON THE
NATURE AND PROPERTIES OF CERAMIC MATERIALS

supported by the

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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COLLEGE OF ENGINEERING
UNIVERSITY OF WASHINGTON



SUMMARY

This report covers Interdisciplinary Research at the University of Washington on the Nature and Properties of Ceramic Materials for the period June 16, 1980 through December 31, 1980. The program is supported by the National Aeronautics and Space Administration through Grant Number NFL-48-004-002. The principal direction of the research is towards improvement in design with brittle (ceramic) materials through studies of design methodology and associated supportive research.

A major supplement to the program for the past year has been the application of brittle design methodologies to the thermal protection system of the Space Shuttle. A complete report on this has been published separately.

The interdisciplinary aspects of the program is evidenced by the participation of 17 faculty and 17 graduate and 7 undergraduate students, representing seven disciplines within the University.

N81-19299#

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INTRODUCTION

This report covers the first half of the fifth year of the interdisciplinary research program at this institution on the nature and properties of ceramic materials with emphasis on design with brittle materials, supported by grant number NFL-48-004-002 from the National Aeronautics and Space Administration. Satisfactory progress has been made towards our stated goals, which are:

1. Advancement of material performance and design methodology as related to brittle materials.
2. Understanding of processing and properties of ceramic materials as related to design requirements.
3. Improvement of communication and understanding both within the University and between academia, government, and industry.

The program is divided into two major components--studies of design methodology and structural ceramics research. The purpose of the former is not only the study, improvement and utilization of probabilistic design but also the development of means for establishing the necessary communication and mutual understanding between the designer and the materials specialist. The research effort consists of interrelated studies of processing, characterization and mechanics, including testing and evaluation. Each of the two major areas are coordinated by a committee composed of faculty members active in that area. Faculty, staff and students participating in this program have been assigned by the University to the Dean of the College of Engineering, who appoints a board of representative faculty members to establish policy and approve the general operation of the program. The composition of the Ceramic Materials Research Committee is shown in Appendix II.

Progress towards the first two goals will be discussed in subsequent sections of this report. A significant impact upon the program resulted from supplemental funding to apply characterization techniques and probabilistic design methods to the thermal protection system for Space Shuttle. Significant progress has been made in this area as discussed in the design methodology section. A separate semi-annual status report was published on this portion of the program in December and was distributed to parties involved in the subject. This effort has had some impact on some aspect of the research portion of this program but these should be minimized during the forthcoming year.

Installations visited by participating faculty during the report period included:

Carnegie-Mellon, University of Pittsburg, Pittsburg, Pennsylvania
 Daimler-Benz, Stuttgart, West Germany
 DFVLR, Cologne, West Germany
 Eaton Corporation, Southfield, Michigan
 Evans Products, Corvallis, Oregon
 Ford Research Lab, Detroit, Michigan
 Imperial College, London, England
 Kaiser Wilhelm Institute, Stuttgart, West Germany
 NASA Headquarters, Washington, D. C.
 NASA-Ames Research Center, Moffett Field, California
 NASA-Johnson Space Center, Houston, Texas
 NASA-Kennedy Space Center, Cape Canaveral, Florida
 NASA-Lewis Research Center, Cleveland, Ohio
 Rockwell International, Inc., Downey, California

Technical meetings attended included:

Pacific Coast Regional Meeting, American Ceramic Society, San Francisco, California
 Glass Division Meeting, American Ceramic Society, Bedford Springs, Pennsylvania
 Highway Vehicle Systems Contractors Coordination Meeting, U.S. Department of Energy, Dearborn, Michigan
 Fracture Mechanics Methods for Ceramics, Rocks and Concrete, American Society for Testing and Materials, Chicago, Illinois
 Century 2 Emerging Technology Conferences, The American Society of Mechanical Engineers, San Francisco, California
 Committee on Nuclear Safety Inspection, Electric Power Research Institute, Palo Alto, California.
 12th National SAMPE Technical Conference, Materials 1980, Society for the Advancement of Material and Process Engineering, Seattle, Washington

The annual review for the NASA technical monitor was held on the University campus November 17-18, 1980. As has been customary in the past, representatives from industry, government and academia were again invited and the following participated:

Charles C. Bersch, Technical Monitor, NASA Headquarters
 Samuel W. Bradstreet, Consultant, Parkland, Oregon
 Joseph C. Conti, McDonnell Douglas Company, St. Louis, Missouri
 Winston Duckworth, Battelle Memorial Institute, Columbus, Ohio
 E. A. Fisher, Ford Motor Company, Dearborn, Michigan
 Alan J. Gnann, Evans Products Company, Corvallis, Oregon
 Glenn W. Hollenberg, Westinghouse Corp., Richland, Washington
 James Keski, Tektronix, Inc., Beaverton, Oregon
 David H. Scharnweber, Eaton Corporation, Southfield, Michigan

Two status briefings were presented on the Shuttle TPS Model program. The first, on September 18, 1980 was given at Rockwell (Downey), to The NASA Chief Engineer. Additional attendees were from, JSC, ARC, Rockwell and Lockheed. The second briefing was given at NASA Headquarters to Shuttle Program Management and OAST personnel on Oct. 8, 1980.

DESIGN METHODOLOGY STUDIES

Coordinating Committee

R. J. H. Bollard, Aeronautics & Astronautics, Chairman
B. J. Hartz, Civil Engineering
A. S. Kobayashi, Mechanical Engineering
W. J. Love, Mechanical Engineering
J. I. Mueller, Ceramic Engineering
W. D. Scott, Ceramic Engineering
R. Taggart, Mechanical Engineering

The major activity in this component of the program has been, again, the offering of the course sequences 476 and 496 at the undergraduate fourth year level and the graduate course 536. The introductory design methodology course, 476, described in detail in the July 1979 status report as the communication development course, and 496, the class design project course, were both covered in the June 30, 1980 report. The follow-on graduate individual project course was successfully carried on through the summer and fall quarters. During the summer period two students studied in detail the continuation of the diesel engine intake valve previously reported under the 496 description and one student took up the prediction of failure of a glass pressurized fluid container of a given shape. The study with the valve resulted in confirmation of the validity of the Weibull statistical model of failure from simple beam MOR data in the prediction of failure in prototype valve specimens subjected to a pressure load on the valve head while supported by an appropriate seat. Further, the summer study focused on the use of compliant layers to accommodate spring collects so that a stem of constant diameter could be used as opposed to the previously reported 496 class design which had a variable diameter stem and required a split valve sleeve. The study was carried out on a computer using a finite element model and indicated that a uniform valve stem and copper compliant layer was feasible. In addition, the design for a test jig was completed. In this jig a total valve incorporating the best of the two previous design studies could be tested to destruction to verify the P.O.F. predictions. This study, in response to a posed problem from an industrial affiliate, is to be continued in the future as additional data on material problems and engine design constraints on such valving system are obtained.

The glass pressure vessel study progressed during this report period to a master's thesis research program and the developments leading to a P.O.F. prediction for the vessel, and identification of the site of most probable failure initiation, is reported in detail in the supporting research section.

The text "Design with Brittle Materials" has passed through a second period of corrections and updating resulting in minor changes. As a workbook in the 476 class it is proving to be invaluable to the students and especially so with their added notes. The book is now available for purchase and while it is not in the category of "best seller" it is finding the expected market in industry and in other institutions planning or already conducting courses on design with brittle materials. And this without any sales promotion since the production is still within the group and designed primarily to serve the immediate course needs.

The listing of industrial problems suited to the 476 models, and 496 projects, continues to grow. It is interesting to note that the two projects begun in the Spring quarter and continued in the Fall, viz., the valve studies and the glass pressure vessels were both selected from this source and with the comment from the students that working on real-life problems was very meaningful to them.

The presentation of company briefings, while continuing, has been necessarily reduced by the dramatic escalation in travel costs. This part of the program is following techniques which hold the promise of being more effective for a given investment in travel. For example, a 3-day workshop is planned for the near future in conjunction with the annual meeting of the American Ceramic Society and a similar presentation later in the year in response to an invitation from a group of European countries.

The final attainment is the publication of an improved version of the general introduction to probabilistic design with brittle materials. The monograph, intended for lower division level students, is also now available for purchase.

This component of the program continues to be a very rewarding aspect of the overall study of ceramic brittle materials for faculty and students alike. The pre-enrollment in 476 for this Winter quarter is at 48, an increase of more than 37% over this last winter. This attests to the application of students in interdisciplinary studies in areas of new technology. The faculty are still enthused with the success of the course and find it a stimulating experience. The university is gradually taking over the support of the courses 476 and 496.

Development of Design Methodology Through Independent Studies

Space Shuttle Thermal Protection System

This project has continued through this reporting period and a second program report has been published. Details of the various areas of inquiry are contained therein. This particular study has also continued to dominate the activities in the program with the understanding of the NASA and in appreciation of the pressing national importance of the area of study.

This same faculty group as identified in the last report continued to operate this particular study, but, naturally, there has been changes in the staff and graduate students involved. The participants and their affiliations in the University are listed below.

Faculty:

- R. J. H. Bollard, Coordinator, Aeronautics and Astronautics
- W. D. Bonow, Engineering, Bellevue Community College
- B. J. Hartz, Civil Engineering
- A. S. Kobayashi, Mechanical Engineering
- A. Komine, Mechanical Engineering
- W. J. Love, Mechanical Engineering
- H. C. Merchant, Mechanical Engineering
- A. D. Miller, Ceramic Engineering
- J. I. Mueller, Ceramic Engineering
- W. D. Scott, Ceramic Engineering
- R. Taggart, Mechanical Engineering
- O. J. Whittemore, Ceramic Engineering

Staff:

- D. W. Gilbert, B. S., Ceramic Engineering
- J. K. Ho, B. S., Aeronautics and Astronautics, B. S., Mathematics
- C. W. Newquist, B. S., Ceramic Engineering
- R. D. Pfaff, B. S., Aeronautics and Astronautics

Graduate Students:

- B. M. Liaw, Mechanical Engineering
- C. Georgiadis, Civil Engineering
- C. F. Chiu, Mechanical Engineering
- A. C. Hansen, Mechanical Engineering
- T. W. Shiu, Mechanical Engineering

Undergraduate Assistants:

- L. L. Glennie, Ceramic Engineering
- D. K. Johnston, Ceramic Engineering
- R. E. Maxell, Ceramic Engineering
- E. S. Tosaya, Ceramic Engineering
- W. L. Vaughn, Ceramic Engineering

The following brief summaries in each of the areas of concentration during this report period give a view of the total effort and an identification of the major findings appearing in the program report No. 2 of September 1980.

- a) Material characterization--Emphasis has been on the behavior of the Nomex strain isolation pad (SIP) in a dynamic loading environment. The apparent growth of the SIP has been recorded and the effect of this growth on the dynamic response of the supported tile observed. The irreversible cumulative damage mechanisms in the SIP have been studied at high magnification as has the bond-line load transfer mechanism from SIP into epoxy and RTV adhesive layers. The effect of different surface treatments on the nylon fiber of Nomex felt has also been investigated.
- b) Mathematical modelling--Experimental evaluation of the conditioning of SIP by different "proof" tests was carried out and the results used in the prediction of the dynamic response of SIP conditioned under moment loading.

The probability of failure (POF) finite element models using Weibull statistical models of failure have been used to predict the tile/SIP/RTV system failure under system loadings recorded during tile array testing. Close correlation between observed failures, for densified and undensified tiles of various geometries, has been demonstrated. This improved technique for prediction of failure over that being used in the design process at this time is to be incorporated in future tile system integrity analyses for predicted mission loadings.

Continuing thermal stress analyses indicate that cracked tile coatings can give rise to high local tensile stresses in the tile material and that, in general, high stresses will occur in the vicinity of tile intrusive attachment devices such as the "Auger".

- c) Dynamic response--Emphasis has been on the response of tiles during time periods representative of typical flight missions and the observation of parametric excitation and the failure rate of the SIP under different mode shape responses. SIP damaged during these tests is being studied at high magnification to gain insight into how the stability of the SIP may be improved. The results of these dynamic tests are being used to evaluate various time-dependent theoretical dynamic models and to provide information necessary to the development of lifetime prediction models.
- d) Crack propagation and lifetime studies--The application of fracture mechanics to bond line and tile body failures is proceeding. There is further evidence to support the models of stable crack growth in the bond line and the existence of critical flaws in the bond line and the existence of critical flaw dimensions in the tile body. Both theoretical and experimental studies are continuing. Data from these studies along with long-term response studies in dynamic load response as part of this project and from data being generated elsewhere, possible lifetime prediction models are being formulated.

Because of the complexity of failure mechanisms in the system and the timewise dependence of the properties of the components these early models are being used primarily to place some bounds on the testing necessary to generate a usable model.

One significant faculty modification was the addition to the group for 2 months during the past summer, of Professor Burnett Bonow, chairman of the Engineering Department at nearby Bellevue Community College. This was deemed a successful experiment to introduce lower division engineering faculty from two year institutions to interdisciplinary efforts and to brittle material design.

Coordinating Committee

W. D. Scott, Chairman, Ceramic Engineering
A. F. Emery, Mechanical Engineering
J. L. Bjorkstam, Electrical Engineering

Objectives

The goal of this portion of the program is to develop an active faculty-graduate student research effort in high performance ceramic materials and structures. This program is to be interdisciplinary through the involvement of faculty and students in several engineering and science departments outside of engineering but remain focused on structural ceramic materials. The research coordination involves facilitating internal and external communications so that the faculty and students are aware of trends and advances in appropriate research areas world-wide. This communication role falls mainly to the Ceramic Engineering faculty as the central point through which flows information on ceramic materials research.

Organization

Proposals for individual research programs are solicited annually by publicity directed throughout the appropriate science and engineering departments. Proposals are reviewed by the Ceramic Materials Research Board, in a setting where the written documents are explained and amplified with a verbal presentation. At the last proposal review, 17 out of approximately 20 proposals were funded. The departments now involved in the research program are Physics, Aeronautics and Astronautics, Mechanical Engineering, Electrical Engineering, Civil Engineering, Metallurgical Engineering and Ceramic Engineering.

Progress

Details of new projects funded this fiscal year were given in the June 30 Status Report. No new projects have been added since that time. The Program Review in November, 1980 provided a focus for substantial participant interaction internally and with visitors. Several participants have attended national conferences dealing with structural ceramics, and visitors have presented seminars in Ceramic Engineering which were attended by representatives from all the participating departments. The Ceramic Engineering Graduate Student Seminar for Autumn quarter, 1980 also concentrated on reviews of current research in structural ceramics.

Future Plans

It is intended to increase the opportunity for direct student-faculty contacts with active research workers through invitations for visits to the University. The annual proposal solicitation and review will be carried out in spring quarter.

OPTO-ACOUSTIC TECHNIQUES FOR LOCATING DEFECTS IN CERAMICS

Faculty Supervisor: John L. Bjorkstam, Professor
Electrical Engineering

Graduate Assistant: Davene Eyres, M. S. Student
Electrical Engineering

Purpose

The purpose of this investigation is to develop an opto-acoustic technique for detecting small ($\leq 20\mu\text{m}$) subsurface (and surface) flaws in materials with high acoustic attenuation. In its final form the research has two major thrusts: (1) the generation of acoustical pulses with high energy density in the spectral range corresponding to wavelengths $\leq 20\mu\text{m}$ (frequencies $\geq 200\text{ MHz}$), and (2) the detecting of acoustical energy scattered from flaws using a method which would be convenient in a production format.

Relevance

This research will lead to improved methods for non-destructive-evaluation (NDE) of structural and electronic ceramics. The technique will have particular applicability to detection and location of small inhomogeneities in materials with appreciable acoustic attenuation and normally smooth surfaces. The experimental methods are closely related to other optical and signal processing experiments underway in the electrical engineering department and, as a result receive from, and contribute to, those other efforts.

Objectives

The primary objectives are, (1) to generate short high intensity, acoustic pulses using differential thermal expansion at a material surface which absorbs the output of a pico second laser pulse train, (2) to detect and quantify the energy so generated in the frequency range $\geq 200\text{ MHz}$, and (3) to develop flexible techniques for defect location which will be applicable to a production line format. Our initial investigations have been directed toward a broad survey of formats for high conversion efficiency and sensitive, calibrated detection methods. For pulse generation we have available a Nd: glass laser ($\lambda = 1.06\mu\text{m}$) which produces a train of ≈ 100 , 5 picosec wide pulses, with 5.7 nanosec spacing, each time the laser is fired. Thus the acoustical pulse train will contain energy at the fundamental (175MHz) and harmonics of 5.7 nanosec interpulse period.

Progress

Difficulties with optical detection have led us to continued pursuit of somewhat more conventional acoustical techniques, while retaining the essential laser pulse generation scheme. To that end we have acquired a quartz transducer, mounted on a buffer rod, and polished for 175 MHz operation. In addition a fixture for use with polymeric piezoelectric detectors has been designed and fabricated. The related electronic systems have been assembled and experiments will be underway very soon.

EVALUATION OF TESTS FOR MECHANICAL PROPERTIES OF CERAMIC MATERIALS

Faculty Supervisor: R. J. H. Bollard
Aeronautics and Astronautics

Purpose

To accurately assess the stress state under given load in specimens commonly used in brittle material characterization and failure tests and thereby assess the errors inherent in such tests and in the use of simple model approximations to the actual stress states. It is intended to extend this assessment to candidate tests not commonly used and to develop error assessments in the correlation of failure results between tests using different specimens, tests using specimens of the same geometry but different size, and, tests of different complexity in the stress field such as between uniaxial and multiaxial states.

Relevance

Since theoretical behavior and failure predictions are based on stress functions and predicted stresses at observed failure, it is apparent that the accuracy of such predictions is directly related to the accuracy with which the stresses can be predicted. For example, in isotropic, homogeneous elastic materials the calculation of stresses and consequent behavior is dependent on the material properties and E and ν and the actual failure stress in a given test. Since the observed failure of brittle material in a given test occurs with a large scatter in values of failure load a statistical model of failure must be used. The most common such model is that due to Weibull and introduces a further material property, σ_0 , and the Weibull parameter m , which is a measure of the scatter in the observed load failure. This model is often refined by the additional parameter σ_u , that stress level below which failure is not observed to occur in a particular test.

In the prediction of failure it is particularly important to know the possible error occurring in the calculated values of σ_c , σ_u and the actual stress state, for in the Weibull model, differences or ratios of these quantities are raised to the power of m and errors are thereby magnified. Furthermore, the accuracy of correlations in failure prediction between tests using specimens of the same geometry but different size, specimens of different geometry, or specimens in which the stress states are different, will be dependent on the accuracy with which the actual stress at failure is predicted. In complicated geometries and loading states these correlations require the knowledge of the stress field throughout the volume of the specimen which fact places further demand on the assessment of error in the derived stress as compared to those actually occurring.

Objectives

To assess errors in the calculation of actual stress states in tests, such as simple beam tests, commonly used in evaluating the so-called modulus of rupture (MOR) for brittle materials; to correlate results of simple beam tests of different geometry and/or size and to other tests with simple stress states, with accompanying error assessment; and to correlate the results of simple stress-state tests of failure to measured and predicted failure in complex stress states with resultant error assessment in the use of simple mathematical models.

Progress:

Statistical models for failure of ceramic brittle materials subjected to biaxial loads have been formulated for the simple case of the edge loaded disc using corrected uniaxial stress failure data. Sensitivity to error in the loading states is being investigated for various disc geometries and constraint conditions as was done for the simple beam in the previous studies. An experimental investigation for alumina discs is being carried out to compare with the various biaxial failure models currently in use. Appropriate corrections are being applied to the disc experiments as well as to the original experimental failure data.

The theoretical and experimental studies are being extended to other biaxial states such as transversely loaded discs, to provide data for prediction of application boundaries for the different failure prediction modes.

STRAIN-RATE SENSITIVITY OF CERAMICS

Faculty Supervisor: I. M. Fyfe, Professor
Aeronautics & Astronautics

Purpose:

The research work in this project is designed to examine the response of ceramics to impact loading, and in particular to determine if shock loading will change the tensile strength properties of the material.

Relevance:

In situations where ceramic materials may be subjected to impact, either by accident or design, it is important to have some idea how the material will respond to this type of environment. In the case of metals, for example, this type of loading can result in improved ductility which is significant in the areas of metal forming and crash worthiness.

Objectives:

The basic objective of this one year project is to develop techniques which will allow ceramics material properties to be determined after the material has been subjected to impact.

Static and dynamic tests have been carried out on internally pressurized ring specimens. The static tests have reached the point where quantitative results can be obtained. The initial design of the dynamic test system was such the rings shattered on impact. This system is being redesigned to try and achieve the goal of an impacted ring that can then be tested statically. When this objective is achieved, it will then be possible to compare the static strength properties of rings that have, and have not been dynamically pre-loaded.

RELIABILITY PREDICTION OF THE STRENGTH OF CERAMIC MATERIALS
UNDER COMPLEX STRESS STATES

Faculty Supervisor: B. J. Hartz, Professor
Civil Engineering

Purpose:

To develop the methodology and data necessary for the prediction of the statistical distribution of the strength of ceramic structural components under complex stress states.

Relevance:

This project requires the utilization and further development of extremely important concepts of materials characterization, particularly under complex stress states, Weibull statistical representation of strength of brittle materials and refined stress analysis via finite element method. A particularly important aspect of this project is the large number of prototypical structural specimens being tested for the determination of the statistical distribution of the structural strength. This project is relevant both to the development of an academic program to teach design for brittle materials and to the needs of industry currently utilizing these design concepts for brittle materials but without the large statistical verification being undertaken in this project.

Objectives:

The objective of the current year is to extend the work completed by Osamu Inoue during the last project period. Primarily this will be an attempt to develop and implement a "Weibull Singularity Element" in the finite element following on the "notch Strength" developed by Inoue which will give the contribution to the "B-integral" in the Weibull P.O.F. approach of an element with a singularity.

Progress:

Because of the enrollment cut in our graduate program it was not possible to get a student assistant on this project summer or autumn quarter, however a research assistant will be available for this project effective January 1, 1981.

The progress during this period consisted of background research by the principal investigator and the completion of the rate controlled loading device for breaking substrate specimens under controlled loading rates to see if this has been a contributing factor in the statistical data collected on substrate strength.

IMPACT DAMAGE

Faculty Supervisor: Albert S. Kobayashi, Professor
Mechanical Engineering

Ashley F. Emery, Professor
Mechanical Engineering

Graduate Assistant: Been Ming Benjamine Liaw, Post MS Student
Mechanical Engineering

Purpose:

The purpose of this project is to explore the feasibilities of establishing phenomenological models of impact damage and erosion of structural ceramics based on dynamic fracture mechanics.

Relevance:

- a) Contribution to Own Discipline: Dynamic fracture mechanics is in its development stage and damage and erosion studies based on dynamic fracture mechanics have not been explored yet. This project is therefore pushing the state-of-the science in fracture mechanics of metals.
- b) Problem Areas: Glass specimens were delivered in August 1980, but the dynamic fracture toughness testing has been delayed due to the dilapidated condition of the megahertz instrumentation in the Department of Mechanical Engineering. Temporary repairs of this outmoded instrumentation has been completed and preliminary check-out of the Fractomat in conjunction with dynamic fracture toughness testing is finally underway.
- c) Progress Toward Current Year Objectives: Fracture dynamic analysis of single edge notched (SEN) specimens, 6.4x25.6mm and 640x2560mm, subjected to uniform end load, uniform extension and fixed end rotation were completed. Dynamic finite element method was used in its generation mode based on an assumed normalized dynamic fracture toughness versus normalized crack velocity relation to estimate the dynamic fracture responses of the SEN specimens. Material properties of silicon nitride, silicon carbide, steel and Homalite-100 plastic were considered. Despite differences in sizes and material properties, the normalized dynamic stress intensity factor versus normalized crack length relations for SEN specimens subjected to a prescribed end loading condition were almost identical. These results verified the contention that scaled modeling with different elastic properties can be used in the dynamic fracture analysis of ceramic fracture specimens provided the normalized dynamic fracture toughness versus normalized crack velocity relations are identical. This finding may have significant influence on the design of future experiments in this project, once the current dynamic fracture characterization is completed.

Objectives:

The first and second year objective is the dynamic fracture characterization of structural ceramics at room through elevated temperatures.

The third year objective is the development of a dynamic fracture model of impact damage.

The fourth and fifth year objectives is the development of an erosion damage model based on the above.

Progress:

- a) Accomplishment: Difficulties in instrumentation has been temporarily corrected and the full scale dynamic fracture testing should be resumed shortly.
- b) Problem Areas: The dilapidated instrumentation in the Solid Mechanics Section of the Department of Mechanical Engineering has repeatedly hindered our research effort. Although temporary repairs are being made, the experimental phase of this investigation may not be completed on schedule.
- c) Progress Toward Current Year Objectives: Only one quarter of the planned research was accomplished.

Paper Presented:

"Dynamic Analyses of Notch Bend Specimens", by A. S. Kobayashi, A. F. Emery and B. M. Liaw at the Committee on Safety of Nuclear Installations International Experts Meeting on Instrumented Precrack Charpy Specimens, Electric Power Research Institute, Palo Alto, California, 1980.

Paper Submitted for Publication:

"Dynamic Analyses of Notch Bend Specimens", by A. S. Kobayashi, A. F. Emery, and B. M. Liaw. To be published in EPRI Report, 1981.

THERMAL SHOCK

Faculty Supervisors: Ashley F. Emery, Professor
Mechanical Engineering

Albert S. Kobayashi, Professor
Mechanical Engineering

Purpose:

The use of ceramics for systems which have to endure hostile environments or which must be subjected to high temperatures, is dependent upon the thermal sensitivity of the ceramic and its resistance to thermal shock. The purpose of the first year of this study was to determine if the current quench test could be used to determine the thermal shock sensitivity of a ceramic material, and if so, how sensitive the test itself was in adequately describing the material sensitivity. The project also aimed to design a test which would be better defined.

Relevance:

Thermal sensitivity characterization is presently done in an approximate way, since the generally accepted quench test is not well defined and because it is difficult to fix the conditions under which the quench specimen undergoes quenching. This project is intended to improve the means for experimentally evaluating the thermal shock sensitivity of ceramics.

Objectives:

The objective is to numerically investigate the behaviour of a ceramic specimen, when subjected to a rapid quenching, in terms of the state of stress induced and the resulting stress intensity factors. The project also aims to develop the 'spot test' which is expected to provide more precise and meaningful ceramic sensitivity results.

Progress:

- A) An analysis was made of a usual quench specimen with temperature dependent properties, and cycling bath temperatures to determine the nature of and the history of possible crack extension. This work is complete and the analytical results have been published. Further work will be concentrated on the continued analytical development of the spot test, and the construction of preliminary test facilities to verify the analysis.
- B) A preliminary analysis of the thermal spot test has been carried out and the results were reported at the Pacific Regional Meeting of the American Ceramic Society. This analysis determined the basic characteristics of the stresses in an uncracked specimen and showed that typical specimen sizes and testing times could be expected to yield thermal shock sensitivity results which are comparable to those determined through the usual quench test.

- C. Further numerical analyses of the experimental tests carried out by Professor A. G. Evans, of the University of California, are being performed. These results will be submitted to the ACS journal as part 2 of the paper describing his form of the spot test.
- C. A survey article of Thermal Stresses and Thermal Stress Induced Fracture is being prepared for publication as a special publication of the ACS.

Papers Submitted for Publication:

"Transient Stress Intensity Factors for Edge and Corner Cracks in Quench Test Specimens", A. F. Emery, A. S. Kobayashi, to appear in the Journal of the American Ceramic Society, 1980.

Degree Recipient:

Mr. Thomas R. Bieler, Master of Science in Ceramic Engineering, Summer quarter, 1980.

Thesis Title:

"The Consequences of Varying Surface Heat Transfer Coefficients, Material Properties and Cyclic Ambient Upon Stress Intensity Factor for Edge Cracks and the Description of Program Intemps, A Finite Element Code".

CHARACTERIZATION OF SILICON NITRIDE BY AUGER
ELECTRON SPECTROSCOPY AND LOW-ENERGY
ELECTRON DIFFRACTION

Faculty Supervisor: S. C. Fain, Jr., Professor
Physics

Graduate Assistant: A. G. Schrott, Ph.D. Candidate
Physics

Purpose:

To study the early stages of the reaction between nitrogen atoms and silicon single crystals and to investigate the structure and composition of silicon nitride surfaces using low-energy electron diffraction (LEED) and Auger electron spectroscopy.

Relevance:

- a) Better physical models appropriate for describing effects of adsorbate-adsorbate interactions and the role of defects and impurities on the early stages of the reaction between nitrogen atoms and silicon surfaces could result from this research.
- b) Improvements in reaction bonding processing might result from understanding the role of surface phases and impurities in nitridation and from a better characterization of the properties of Si_3N_4 surface layers.

Objectives:

- a) Understand the interactions between the reacting atoms and the effect of these interactions and of defects and impurities on the reaction rate and surface phases formed during nitridation of silicon single crystals in ultra-clean environment where LEED Auger surface characterization can be used.
- b) Assess the usefulness to reaction-bonding process optimization of Auger electron spectroscopy analysis of surfaces obtained by fracture of test samples in ultra-high vacuum.

Progress:

- a) Annealed Si (111) crystals have been reacted at $T < 1050^\circ\text{C}$ with nitrogen atoms produced by electron dissociation of N_2 gas near 10^{-4} Torr. The initial stage of the reaction is rate limited by the incident nitrogen flux, has a constant sticking coefficient of order 1, and produces a well-ordered "quadruplet" LEED pattern for $T \approx 1050^\circ\text{C}$ which becomes more intense as the $\text{Si}(7 \times 7)$ spots fade away. With the disappearance of the $\text{Si}(7 \times 7)$ pattern, the reaction proceeds much more slowly, is apparently rate limited by diffusion, and produces a poorly ordered LEED pattern

which gradually replaces the "quadruplet" pattern. The unreacted Si Auger peak shifts to lower energy during the initial stage and disappears during the second stage at about the same time as the N Auger peak saturates. The layer thickness at saturation is estimated to be about 8-10Å.

The surface nitride layers can be removed by heating the crystal to about 1200°C. Preliminary measurements of the desorption products have been made with a borrowed quadrupole mass spectrometer. These measurements have been very useful for identifying desorption products and providing new information about different stages of the reaction.

Aluminum and iron are believed to affect the equilibrium between α and β phases in the nitridation process. Our previous attempts to study the role of Fe in the initial stages of nitridation of Si single crystals presented the following difficulties: i) the presence of contaminants, ii) iron seems to diffuse into the bulk at the temperatures used to nitride our samples. Aluminum evaporation is easier to achieve with a low degree of contamination. Previous work (in other laboratories) showed the feasibility of studying the effect of adsorption of Al on Si single crystals at temperatures up to 1000°C using surface techniques. An electron beam evaporator for aluminum has been designed and is presently being tested.

- b) Measurements on fracture surfaces of RBSN material have been temporarily delayed.

CHARACTERIZATION OF SILICON NITRIDE BY EXAFS

Faculty Supervisor: E. A. Stern, Professor
Physics

Graduate Assistant: C. Bouldin, Ph.D Candidate
Physics

Purpose:

We propose to identify and characterize the surface properties of Si_3N_4 that are important to the sintering processes by which this material is produced. This will be done by the coordinated application of two different probes of the surface properties: adsorption isotherm and extended x-ray absorption fine structure (EXAFS) measurements. The isotherms will be used to identify the population distribution of different types of adsorption sites characterized by their differing adatom binding energies. The microscopic structure of these sites on an atomic scale can then be determined by the EXAFS measurements. Studies of samples produced under varying conditions allow us to correlate the microscopic surface properties with sintering parameters.

Relevance:

At present, detailed knowledge on an atomic scale of the microscopic physical processes involved in sintering is not available. The present work is aimed at developing such knowledge for one particular material, Si_3N_4 , but with the expectation that much of the same knowledge can be carried over to other materials. Once the physical basis of the sintering process is better understood, then this knowledge can be applied to optimizing the fabrication of technologically important products. In the case of Si_3N_4 , these include components exposed to high temperature environments such as high performance gas turbines and combustion chambers. In the past, much of this optimization has been on a trial and error basis.

Objective:

Objectives for this year were to make measurements of the iron impurities in silicon nitride at many stages of the processing and under variable conditions of iron concentration and production techniques. Also, measurements were to begin on Kr adsorbed on silicon nitride to study the surface properties of silicon nitride by observing the EXAFS of the Kr edge.

Progress:

The iron studies have been completed and the data is being analyzed. The Kr studies have not yet been given beam time by SSRL although considerable work has been done preparatory to making the Kr measurements.

The technical challenge presented by the Kr measurements have given impetus to the development of EXAFS techniques by our group. First of all, the adsorbed Kr samples are extremely dilute and this has caused continued refinement of sophisticated detection techniques to deal with the high dilution.

Also, we have recently completed a laboratory ESAFS facility that allows us to make many of our Fe edge measurements here. We are presently working to extend the energy range of this facility to include the Kr edge as well.

Iron edge measurements from a full production run of silicon nitride have been made at various stages in the processing from the starting powder, which had Fe deliberately added to a level of ~1.5 wt%, up to the final nitrified product. It was observed that, as successively higher temperatures were encountered, the Fe was gradually converted from its initial form of Fe_2O_3 to an iron silicide which is very similar to FeSi_2 . The principal result of these measurements is that the Fe impurities are gradually converted to an iron silicon form as the temperature is increased. This conversion is substantially completed after the sintering stage of the processing. The temperature needed to convert the iron oxide to an iron silicide seems to be about 1000-1200°C. Our previous measurements showed that the Fe impurities in milled silicon powder start in a silicide form.

Finally, we have completed analysis of Kr adsorbed on Grafoil for coverages of .10, .20 and .35 monolayers. This analysis shows that we can clearly distinguish different sites on the surface. This work is currently being written up for publication.

RESEARCH ON HIGH PURITY MATERIALS (Si_3N_4 and SiC)

Faculty Supervisor: A. D. Miller, Associate Professor
Ceramic Engineering

Graduate Assistant: C. W. Newquist, Research Engineer
Ceramic Engineering

Purpose:

The purpose of this project is to:

1. Establish base line data on processing with smallest possible impurity levels.
2. Understand the role of additives, if they are really necessary, in various kinds of processing.

Fine grained compacts of high purity silicon will be nitrided and the progress of the nitriding process observed by ceramography. The amount and character of porosity as well as of unreacted silicon will be measured and the mechanical properties of high purity reaction bonded Si_3N_4 measured.

The optimum grain size and distribution will be postulated and specimens made to optimize the structure. This may well involve bi or tri-modal distribution to optimize density as well as to control the reaction bonding characteristics.

Relevance:

By studying the behavior of high purity materials as a base-line, the role of additives is made much easier to understand. Thus processes may be optimized to improve performance and allowed more detailed trade off of properties versus process cost and applications requirements.

Objectives:

The objective for the current year is to identify the role of iron as an additive to silicon as a sintering aid. This will be accomplished with assistance from other projects in the program.

Progress:

1. Fabrication of Si_3N_4 setter plates for use in nitriding test specimens, and Si_3N_4 crucibles for nitriding loose powders has been completed.
2. High purity silicon powder (99.999% Si, -100 mesh) has been obtained and will be jet attrition milled.

3. Si_3N_4 plates and discs have been prepared from ball milled metallurgical grade silicon (0.6% Fe) for MOR test bars and x-ray diffraction specimens.
4. SEM analysis of the Si_3N_4 fiber matte that occurs on the surface of nitrided silicon compacts shows uniform fiber diameters of 1000-2000Å. Edax analysis of unbroken fiber ends shows no evidence of iron concentration as would be expected from the VLS growth mechanism, and spherical beads at the fiber ends were not readily apparent. The LD aspect ratio of the intertwined fibers was generally much greater than 400/1, with occasional tree-like branching into clusters of short limbs. This $\alpha\text{-Si}_3\text{N}_4$ surface matte is more abundant on material containing 0.6% Fe (no Fe added) than on material containing 3.6% Fe (3% Fe added as Fe_2O_3).
5. Rapid thermocouple degradation in the brew nitriding furnace is a persistent problem and various solutions are being pursued.

OXIDATION RESISTANCE OF SILICON NITRIDE

Faculty Supervisor: William D. Scott
Ceramic Engineering

Graduate Assistant: D. Choi, M. S. Student
Ceramic Engineering

Purpose:

To study the microstructure and morphology of SiO_2 surface layer formed on Si_3N_4 by oxidation. To measure the time and temperature relationships for the formation of amorphous and crystalline silica films and the stability and coherence of these films relative to the underlying material microstructure.

Relevance:

Silicon nitride is proposed for applications in high temperature oxidizing conditions for many hundreds to thousands of hours with frequent temperature cycles. The SiO_2 layer formed, while essential for chemical stability, is not insignificant in terms of mechanical fit, contact surfaces, and simple thermal spalling.

Objectives:

First Year: To investigate the formation of SiO_2 films on CVD silicon nitride and on a well characterized reaction bonded silicon nitride at 1000 and 1300°C.

Second Year: To relate the morphology of the SiO_2 protective layer to the morphology of the underlying silicon nitride which has various densities and pore configuration.

Progress:

a. Polished CVD silicon nitride specimens have been oxidized for 24 hours at 1000 and 1300°C. In the first case, an amorphous SiO_2 layer was formed with thickness as measured by interferometry of about 0.05 micrometers. At 1300°C, well defined cristobalite crystals were developed. These were identified microscopically and by x-ray diffraction. The thickness of this layer was estimated to be between 2 and 10 micrometers.

b. Reaction bonded silicon nitride samples have been received from Ford Motor Company, and are now being polished in preparation for oxidation along with CVD material. No particular problems have arisen in the first 3½ months of this study.

EFFECT OF FLAWS ON BRITTLE FAILURE

Faculty Supervisor: R. Taggart, Professor
Mechanical Engineering

James I. Mueller, Professor
Ceramic Engineering

Graduate Assistant: Bruce Zornes, M. S. Student
Ceramic Engineering

Purpose:

Analysis of the effect of flaws on the successful performance of engineering structure designed with brittle materials, specific case considered- a current commercial soft-drink one-way bottle design.

Relevance:

This is a basic design problem to Mechanical Engineering. It utilizes FEM stress analysis iteration to minimize stresses and to decrease the POF based on measured material Weibull parameters leading to the development of improved design correlating with changes in processing.

Progress:

A detailed computer-aided study of the stresses arising from geometrical constraint and internal pressure loadings for given design configuration has been prepared for a current commercial soft-drink one-way glass bottle. A minimum-stress design profile was accomplished. The accumulation of a large statistical data base for the characterization of the glass material in terms of Weibull parameters is near completion.

It is contemplated that the completion of the data-base accumulation, correlation of the calculated Weibull parameter with failure predictions of the glass structure under load, and finally testing to failure of the actual bottle test population will be accomplished in the next six month period.

LATTICE DEFECTS IN Si_3N_4 and SiC

Faculty Supervisor: T. G. Stoebe, Professor
Metallurgical Engineering

Graduate Assistant: M. S. Donley, M. S. Student
Metallurgical Engineering

Purpose

The purpose of this research program is to provide a basis for the understanding of the influence of lattice imperfections on the observed physical properties of Si_3N_4 and SiC . Paramagnetic impurities are of particular interest here, and the principal technique used in this study is electron spin resonance (esr).

Relevance

Although iron is a common impurity in the Si used to manufacture Si_3N_4 , the role of iron impurities in the processing and properties of Si_3N_4 is not well understood. ESR provides an excellent method for the following types of lattice sites occupied by paramagnetic Fe^{3+} ions since the observed esr signals are characteristic of the Fe^{3+} nearest neighbor ions. Changes in these signals during processing provide the possibility of following the location of the Fe^{3+} iron and of determining its importance in the processing steps used. In combination with studies of iron impurities being undertaken in the Physics department using Auger surface analysis and EXAFS studies of bulk iron in Si and Si_3N_4 this provides means for a complete understanding of the role of iron in Si_3N_4 processing.

This is a new project, initiated in July 1980 current year objectives and preliminary results to date are discussed below.

Objectives

The principal objective of this year's work is to determine those lattice sites occupied by iron impurity ions in Si and Si_3N_4 and to follow changes in these sites during nitriding and sintering processes in this system.

Progress

Preliminary investigations in Si and Si_3N_4 prepared from iron-containing starting materials show two types of esr absorption lines. One is a sharp line characteristic of Fe^{3+} free in the lattice; the other is a broad line characteristic of Fe-Fe interactions in a precipitate phase such as iron silicide. Both are seen in several different forms in metallurgical grade Si, in ball milled and leached Si, in reaction bonded Si_3N_4 and in pressed and sintered Si_3N_4 .

Principal problem area in this program has been related to the esr spectrometer, which has worked only erratically since September. Use of another machine has been obtained until repair is accomplished.

Further investigations are planned utilizing materials on order from several suppliers. Once a complete analysis of the observed esr signals is accomplished, a series of experiments monitoring Fe^{3+} step-by-step during processing will be undertaken.

FAILURE ANALYSIS

Faculty Supervisor: A. D. Miller, Associate Professor
Ceramic Engineering

Graduate Assistant: E. Savrun, Ph.D Candidate
Ceramic Engineering

Purpose:

The purpose of this project is to assess the relationship between microstructural features and failure behavior for brittle materials of interest.

Relevance:

Precise understanding of the microstructural features accompanying failure would be very useful for developing structural ceramics whose fracture sensitivity is reduced. Such information would be important not only to the materials community, but also other disciplines participating in design with brittle materials, such as aeronautical, civil, and mechanical engineering.

Objectives:

- A. Develop a facility and expertise necessary for examination and interpretation of failed brittle material test specimens. (Included in this is the careful review of work by other investigators, and verification of our work by these investigators.)
- B. Examine and determine the relationship between microstructural features and failure behavior for test specimens generated elsewhere and at this University, particularly those from projects under NASA sponsorship.
- C. Establish a method for reporting fractography results, and compile these results into a single or multi-volume publication on fractography of brittle materials.

Progress:

Work continued on characterization of fracture surfaces on injection molded RBSN MOR bars. The fractures in these bars were typically associated with microstructural features such as large pores or groups of pores near the tensile surface of the specimen. Surface flaws due to specimen preparation appeared to be less important in these specimens. The analysis of RBSN fracture surfaces is complicated by the fine grain size of the specimens and by the large amount of fine porosity. Another study was conducted on fracture surfaces generated in aluminum oxide ring burst specimens subjected to radial

loading. Two conditions were used in loading the test specimens, (1) a slow loading by simply increasing fluid pressure in a bladder internal to the ring specimen and (2) shock loading by an exploding wire through which very large electrical currents were passed. The tests were conducted at room temperature. These specimens showed no indication of single fracture origin but rather multiple origins. The fracture surfaces showed no significant difference, although plastic flow lines were somewhat more evident in the slowly loaded specimen. In particular, no evidence was seen of a general intergranular fracture behavior in one specimen versus transgranular in the other.

The search still goes on for a suitable format for displaying and cataloging fracture analyses of brittle specimens.

APPENDIX I

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APPENDIX III

Papers Presented:

"Dynamic Analyses of Notch Bend Specimens", by A. F. Emery, A. S. Kobayashi and B. M. Liaw. Presented at CNSI Meeting, Electric Power Research Institute, December 1980, Palo Alto, California.

"An Interdisciplinary Program in Design with Brittle Materials", W. J. Love, R. J. H. Bollard, B. J. Hartz, J. I. Mueller, W. D. Scott, R. Taggart and O. J. Whittemore. Presented at International Conference on Reliability, Stress Analysis and Failure Prevention, Century II Emerging Technology Conference, August 1980, San Francisco, California.

"Design with Brittle Materials an Interdisciplinary Educational Program", by J. I. Mueller, R. J. H. Bollard, B. J. Hartz, A. S. Kobayashi, W. J. Love, W. D. Scott, R. Taggart, O. J. Whittemore. Presented at 12th National SAMPE Technical Conference, Materials, 1980, Society for the Advancement of Material and Process Engineering, October 1980, Seattle, Washington.

Papers Published:

"Design with Brittle Materials an Interdisciplinary Educational Program", by J. I. Mueller, R. J. H. Bollard, B. J. Hartz, A. S. Kobayashi, W. J. Love, W. D. Scott, R. Taggart, O. J. Whittemore. Proceedings from 12th National SAMPE Technical Conference, Volume 12, MATERIALS 1980.

"Design Analysis of An Automotive Exhaust Gas Sensor", by F. D. Gac, T. R. Bieler, J. P. Chakraverty, J. T. Ewanich, C. Georgiadis, O. Inoue, D. McLaren and R. Taggart. Ceramic Engineering and Science Proceedings, July-August, 1980.

"An Interdisciplinary Program in Design with Brittle Materials", W. J. Love, R. J. H. Bollard, B. J. Hartz, J. I. Mueller, W. D. Scott, R. Taggart and O. J. Whittemore. Proceedings of Century II Emerging Technology, August, 1980.

Papers Submitted for Publication:

"Dynamic Analyses of Notch Bend Specimens", by A. S. Kobayashi, A. F. Emery and B. M. Liaw. To appear in EPRI Report, 1981.

Thesis Titles:

"The Consequences of Varying Surface Heat Transfer Coefficients, Material Properties and Cyclic Ambient Upon Stress Intensity Factor for Edge Cracks and the Description of Program Intemps, A Finite Element Code", Mr. Thomas R. Bieler.

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